



**Polypropylene**  
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**16**

# **Composites Made of Polypropylene Nonwoven Fabric with Plasmas Layers**

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## **1. Introduction**

Engineering of materials used for shielding from electromagnetic fields is currently one of the most extensively developing field of applications of composite materials (Bula et al., 2006; Jaroszewski & Ziaja, 2010; Koprowska et al., 2004, 2008; Sarto et al. 2003, 2005; Wei et al., 2006; Ziaja et al., 2008, 2009, 2010). The choice of suitable materials for the shields and their appropriate arrangement have an essential meaning. Development of lightweight and resistant to environmental exposure shielding materials is possible by using substrates of polypropylene and plasma technology (Ziaja&Jaroszewski, 2011).

The shields for suppression of electric field were made in the form of composites of polypropylene unwoven fabrics with deposited plasma layers. Additional advantage of the application of the method is the possibility of plasma cleaning of a fabric surface and modifying its surface properties. The unique properties of pulse plasma make possible to obtain metallic and dielectric coatings on polypropylene fabrics, which are not achievable by standard methods. The coatings are characterized by a good adhesion to the substrates.

The surface of the samples was examined in two ways: by metallurgical microscope Nikon MA200 and scanning microscope Quanta 200 in the low vacuum mode. To identify the structure of the obtained layers the X-Ray radiography was used. Additionally properties of the composites was studied using impedance spectroscopy. The method of impedance spectroscopy allows one to connect the measured frequency characteristics with the physical structure of tested material and the alternations in the structure. This method has been used by the authors to determine the properties of plasma layers deposited on a polypropylene nonwoven fabric (Jaroszewski et al., 2010a; Pospieszna et al., 2010; Pospieszna et al., 2010b).

## **2. Polypropylene nonwoven fabric with plasma layers in EM technique**

Polypropylene materials (PP), because of their electric properties (such as surface resistivity  $\rho_s$ , volume resistivity  $\rho_v$ , dielectric loss factor  $\tan\delta$ , permittivity  $\epsilon$ ), mechanical properties and resistance to noxious agents (resistance to acids, bases, salts and organic solvents) are used in various industries. Polypropylene materials characterise, also, with the lowest specific density among widely used polymers. Those properties predispose polypropylene to be used as a substrate for composite protective screens shielding people and electric or electronic devices against noxious activity of electromagnetic (EM) fields. Composite shields

are fabricated through metallizing film surfaces or PP nonwoven fabric. Metallic layer thickness does not exceed several micrometres. One or several types of metal as well as conductive metal oxides can make up a metallic layer. Due to their lightness and mechanical strength, those composites are an alternative to classic EM field shielding materials.

Due to characteristic surface properties, PP is a very difficult material to metallize. Ideal for that purpose is the magnetron sputtering method described in papers (Ziaja&Jaroszewski, 2011; Ziaja et al., 2010; Ziaja et al., 2008). Obtained coefficients of shielding effectiveness (SE) of popular composites based on PP/Me matrixes exceed up to 60dB (Me=Zn SE exceeds 60 dB, Me=Cu SE approx. 35 dB, Ti approx. 30 dB).

SE does not depend solely on type of material and its surface and volume resistivity, but also on fabrication (crystal structure) of applied layers. Crucial for SE value is not only the number and resistivity of conductive bridges forming on nonwoven fabrics' surface, but also the specific surface area of applied layers. The more expanded the surface the higher the SE value. Therefore, in order to evaluate those composites' suitability for electromagnetic field shielding screens – apart from electric properties – surface morphology of applied layers has to be known.

### 3. Morphology of PP plasma composites

As means to determine crystal structure of layers, x-ray diffraction method was used. Surface morphologies and chemical composition was determined by a scanning microscope equipped with an x-ray microprobe.

Phase composition of samples was analysed by means of x-ray examination carried out on DRON-2 diffractometer producing Fe filtered Co radiation of  $\lambda = 1.7902 \text{ \AA}$  wavelength. Scanning was carried out according to the wide angle x-ray scattering method at  $\Delta 2\theta = 0.05^\circ$  spread with scattering angle  $2\theta = (40 \div 65^\circ)$ . Diffraction patterns were analysed by the Xrayan programme through comparison of interplanar d-spacing of reflection intensity I to PDF files data.

Morphology analysis of PP/Me composites' surface was carried out using the VEGA/SBH scanning electron microscope manufactured by TESCAN. The microscope is intended for scanning conductive samples in a high-vacuum chamber. At 30kV voltage the maximum resolution is 3.0 nm. Magnification ranges between 6-1000 000 times at specimen current from 1pA to 2uA. The electron optical column is composed of four lenses enabling smooth system configuration and scanning at an optimum resolution. The microscope is additionally equipped with an EDS system attachment [scattered electrons energy analysis] INCA PENTAFTx3 of 133 eV resolution. One of system parts is an analyser enabling point to line analysis of samples' chemical composition.

Structural and electric properties of PP/Me composites depend on magnetron sputtering process parameters (current density at sputtered electrode, pressure and composition of working gases, distance between nonwoven fabric and the target, deposition rate ). By changing those parameters, chemical composition and structure of deposited layers can be manipulated, thus their electrical parameters can be modified. Usually, layer thickness is adjusted by changing gun power or deposition rate. X-ray radiography examination of layers deposited at the same rate, but at increasing power emitted on sputtered electrode are

characterised by increased intensity of characteristic lines. Examples of PP/Zn composites' x-ray spectra were presented in fig. 1. One can note, that at low gun power obtained Zn layers are half-amorphous, indicative of which is lack of Zn-characteristic reflections (fig. 1a). EM field shielding effectiveness is low and does not exceed 10 dB. By increasing the power, increased are not only the layers but their crystallisation as well, causing conductive bridges to form (fig. 1b, 1c, 1d). Surface conductivity decreases and coefficient of shielding effectiveness ranges between 10-30dB. Further power increase induces more conductive bridges of expanded specific surface area to form.

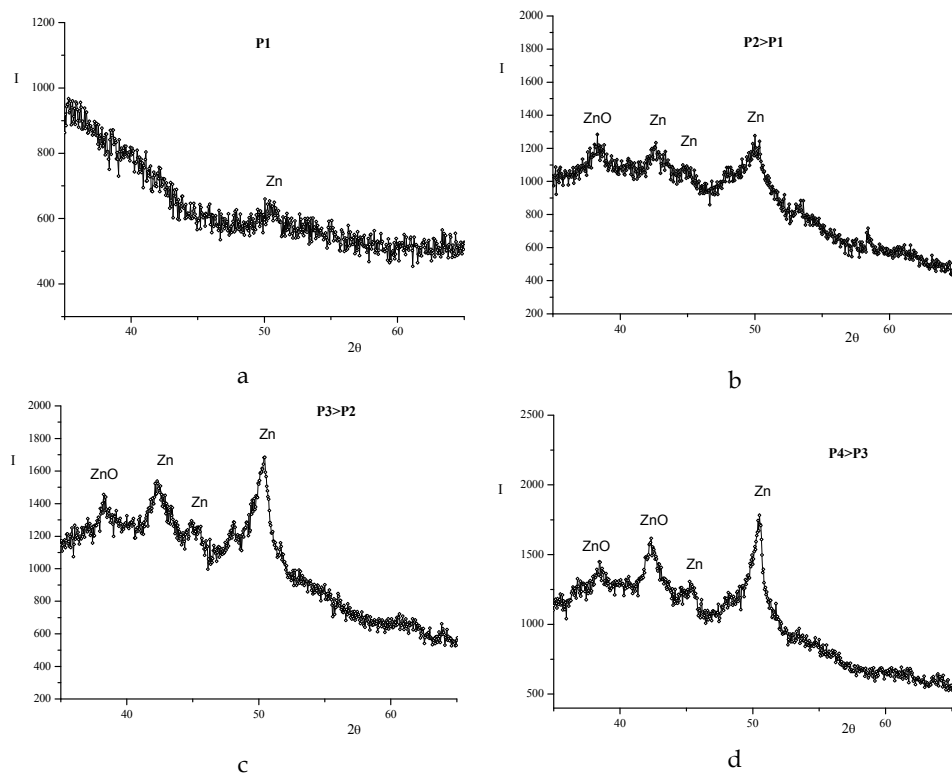
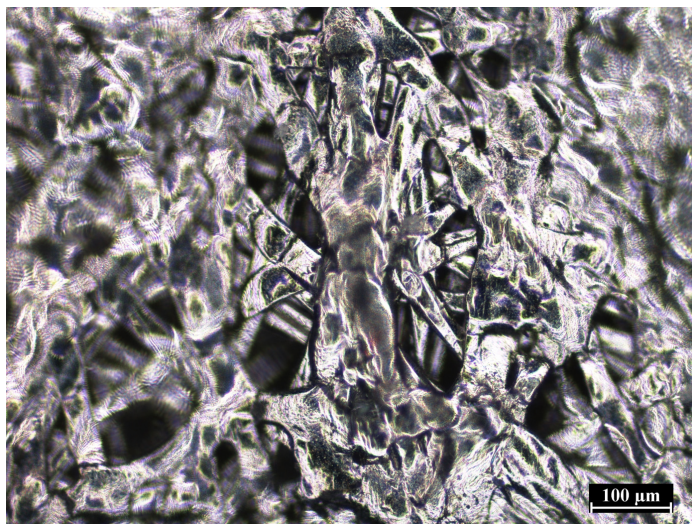


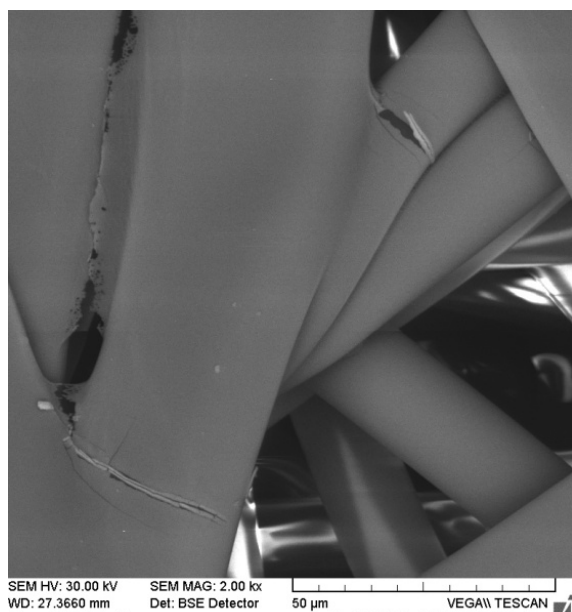
Fig. 1. X-ray radiography spectra of zinc layers deposited on polypropylene nonwoven fabric at different emitted power (P1, P2, P3, P4)

Not only surface fibres are coated with metal (fig 2a), but also the nonwoven fabrics' internal fibres (fig. 2b) and areas between fibres (fig. 2c). Similar crystallization are observable for zinc layers deposited on polypropylene film (fig. 3). A layer obtained in that manner characterises with large specific surface area, which disperses electromagnetic field and increases shielding coefficient SE at the same time. Cross-section of such composite is presented in Fig. 4. It comes to one's attention that metallic layers on fibre surface are solid and uniform. Best composites characterise with SE~ 60 dB, which not only stems from low surface resistivity, but also from expanded surface of metallic layers (fig. 5).

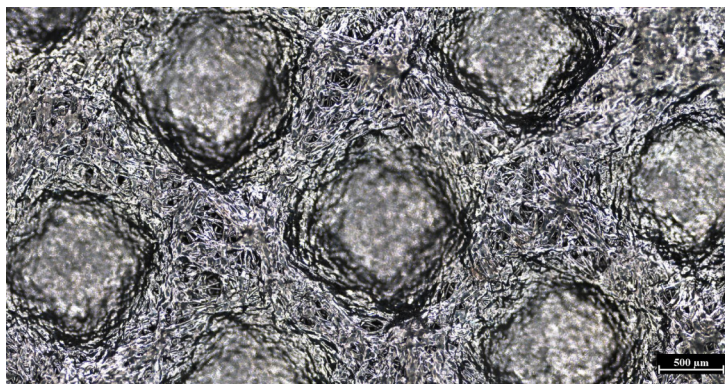
Similar results are observable for other metallic layers, e.g. Al, Ti. Higher layer crystallisation and tighter texture are also notable. Reference book (Ziaja&Jaroszewski, 2011) discusses the method of crystallising Ti layers.



a



b



c

Fig. 2. Surface morphology of Zn layers deposited on polypropylene nonwoven fabric PP Film

Layers deposited on PP films display different behaviour. Layers obtained at highest gun powers remain amorphous. At identical layer deposition parameters the materials characterises with higher SE than the nonwoven fabric. It stems from continuous structure of layers deposited on film as opposed to conductive mesh formed on the surface of nonwoven fabrics. As in case of Ni, Fe or Al layers, SE of film is 10 dB higher.

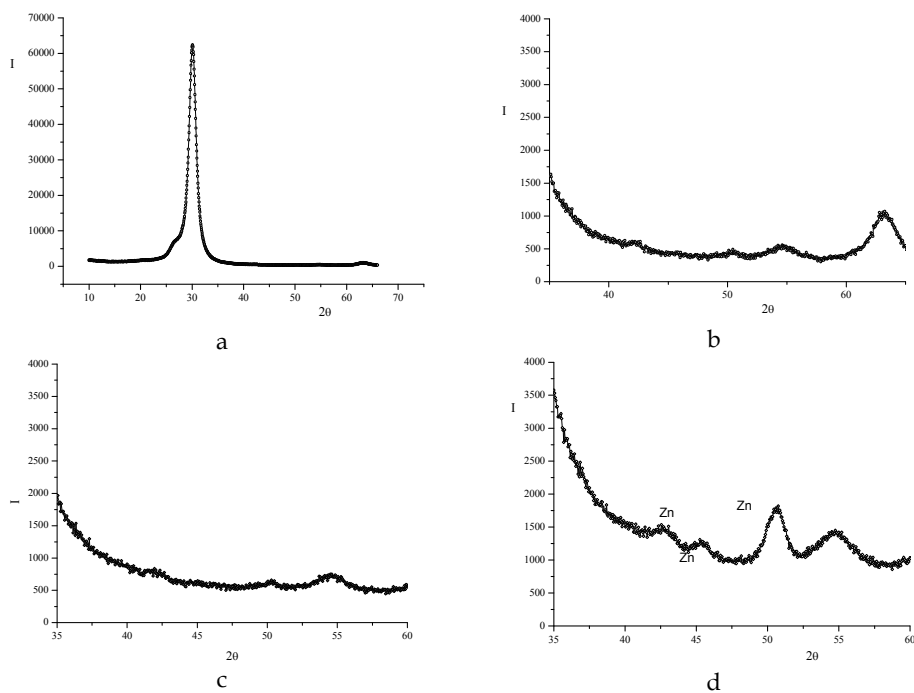


Fig. 3. X-ray radiography spectra of zinc layers deposited on polypropylene film at different emitted current densities

Similar relations in samples' morphology, were noticed by authors in composites with carbon plasma layers deposited on PP nonwoven fabric. They are still, however, characterised by lower SE compared to composites with metallic layers. Likewise promising results of using carbon layers were presented (Wang et al., 2011), where C layers were deposited by silk-screen printing. Presented layers were in form of short and long nanotubes, whose length was critical for SE.

Based on the above-mentioned, nonwoven fabrics with layers are an eligible alternative for classic shielding mats.

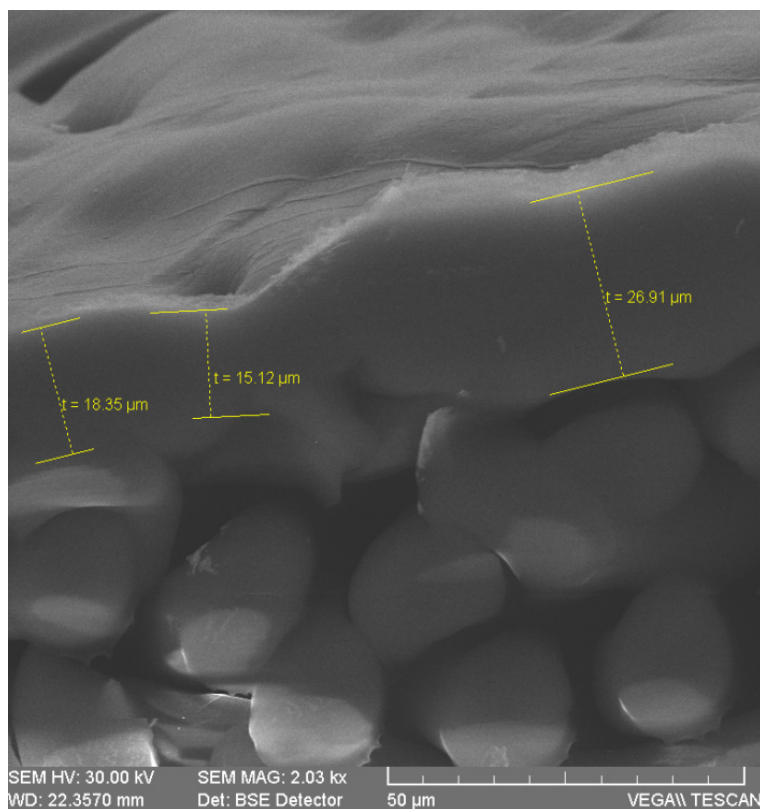


Fig. 4. Cross-section of zinc layers deposited on polypropylene nonwoven fabric



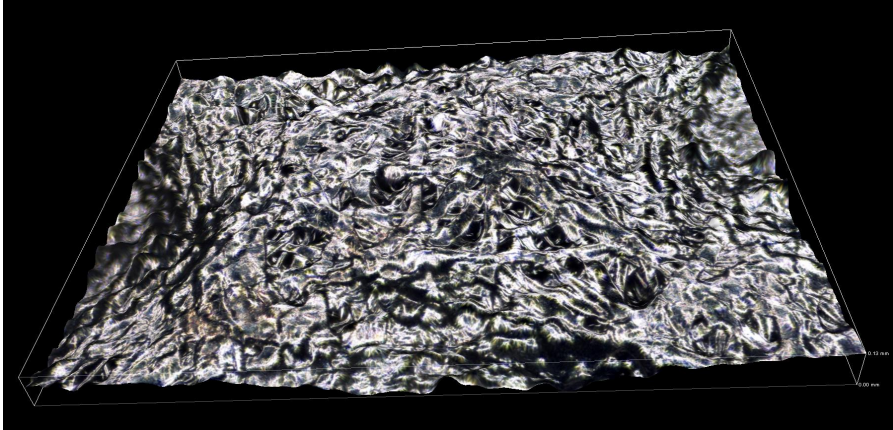


Fig. 5. Expanded surface of metallic layers deposited on polypropylene nonwoven fabric

#### 4. Determination of composites dielectric properties by impedance spectroscopy

Impedance spectroscopy is a modern and exceptionally effective tool for analysing different materials, including complex composites systems. The method draws on measuring linear electric answer of examined material to excitation in form of low amplitude, sinusoidally alternating voltage  $u(t) = U_m \sin(\omega t + \psi_u)$ . The voltage applied to the electrode system, between which examined specimen is placed, induces sinusoidal current  $i(t) = I_m \sin(\omega t + \psi_i)$  to flow through the sample with effective current  $I$ , at phase displacement by angle  $\varphi = \psi_u - \psi_i$ . Analysing that answer in wide spectrum of frequencies yields useful information on conductivity and polarisation phenomena taking place in the examined material.

In the field of frequencies, spectral transmittance  $\underline{H}(\omega)$  is usually used for linear circuits to describe electrical answers. It is defined as a relation of complex responses  $\underline{Y}$  and excitation  $\underline{X}$  of investigated circuit:

$$H(\omega) = Y/X = Y e^{j\psi_y} / X e^{j\psi_x} = Y/X e^{j(\psi_y - \psi_x)} = Y/X e^{j\varphi} \quad (1)$$

where  $\psi_y - \psi_x = \varphi$ .

Its module

$$|\underline{H}(\omega)| = Y/X \quad (2)$$

and argument

$$\varphi = \varphi(\omega) = \arg \underline{H}(\omega) \quad (3)$$

are known as amplitude and phase characteristic of spectral transmittance.

In impedance spectroscopy, spectral transmittance  $\underline{H}(\omega)$  usually has a form of either complex impedance  $\underline{Z}(\omega)$  or complex admittance  $\underline{Y}(\omega)$ .

For a given two-terminal network, complex impedance is defined as relation of complex voltage  $\underline{U}$  values and that voltage-induced current  $\underline{I}$ :

$$\underline{Z}(\omega) = \frac{\underline{U}(\omega)}{\underline{I}(\omega)} = |Z(\omega)|e^{j\varphi(\omega)} \quad (4)$$

$$\underline{Z}(\omega) = \text{Re}\underline{Z} + \text{Im}\underline{Z} = R + jX \quad (5)$$

where  $\text{Re}(\underline{Z})$  and  $\text{Im}(\underline{Z})$  are real and imaginary parts of complex impedance (resistance  $R$  and reactance  $X$ ).

Expression describing complex admittance becomes:

$$\underline{Y}(\omega) = \frac{1}{\underline{Z}(\omega)} = \frac{\underline{I}(\omega)}{\underline{U}(\omega)} = |Y(\omega)|e^{-j\varphi(\omega)} \quad (6)$$

$$\underline{Y}(\omega) = \text{Re}\underline{Y} + \text{Im}\underline{Y} = G + jB \quad (7)$$

where  $\text{Re}(\underline{Y})$  and  $\text{Im}(\underline{Y})$  are real and imaginary part of complex admittance (conductance  $G$  and susceptance  $B$ ).

In practice, the examined sample can be assigned with an equivalent electrical model in form of either parallel or serial connection of resistor and capacitor (Fig. 6), which can be specified by relevant real and imaginary parts of investigated transmittance (admittance and impedance). Assumed model enables to separate active and passive current parts, in case of parallel equivalent of the sample (Fig. 6a) and voltage – in case of serial model (Fig. 6b).

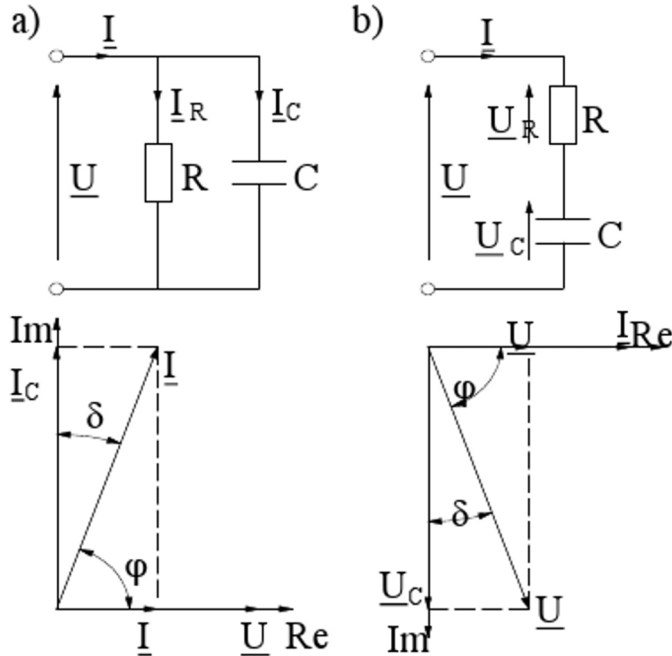


Fig. 6. Capacitor equivalent circuit with real dielectric phase vectors: a) parallel circuit, b) serial circuit.



In equivalent electrical model in form of parallel connection of resistance  $R$  resistor and ideal capacitor of  $C$  capacity, the active current part  $I_R = U / R$  ( $U$  voltage phase) and passive  $I_C = j \omega C U$  (voltage lags the current by  $\pi/2$  phase) represent leakage current and capacitor charging current respectively. Complex admittance measured for that circuit can be represented by:

$$\begin{aligned} \underline{Y}(\omega) &= \underline{I}(\omega) / \underline{U}(\omega) = (\underline{I}_R + \underline{I}_C) / \underline{U} = 1 / R_p + j \omega C_p = \\ &= G_p + j \omega C_p = j \omega (C_p + G_p / j \omega) = j \omega (C' - j C'') = j \omega \underline{C} \end{aligned} \quad (8)$$

where

$$\underline{C}(\omega) = (C' - j C'') = \underline{Y}(\omega) / j \omega \quad (9)$$

are defined as complex capacity and  $C'$  and  $C''$  are its real and imaginary parts respectively.

Complex material parameters can be determined using impedance measurement of simple test structures of given electrode geometry and dimensions of specimen (distance  $d$  and electrode surface  $S$ ) and equivalent complex admittance circuit. Such material parameter is complex conductivity  $\sigma(\omega)$ , which is obtained by multiplying two-terminal network's complex admittance expression by the  $d/S$  parameter:

$$d / S (G_p + j \omega \underline{C}_p) = \sigma' + j \omega \varepsilon_0 \varepsilon_r = \sigma'(\omega) + j \sigma''(\omega) = \underline{\sigma}(\omega) \quad (10)$$

where:

$\sigma'$  – material conductivity,

$\varepsilon_0$  – vacuum permittivity,

$\varepsilon_r$  – relative permittivity of the material,

Its real and imaginary parts are  $\sigma'$  and  $\sigma''$  respectively.

Introduction of complex capacity  $C$  requires introduction of the notion of complex relative permittivity  $\varepsilon$ , to which it is proportionate by definition:

$$\underline{C}(\omega) = C_0 \underline{\varepsilon} = C_0 (\varepsilon' - j \varepsilon'') \quad (11)$$

where:  $C_0$  – geometric capacitance of the electrode system, real part  $\varepsilon'$  and imaginary part  $\varepsilon''$  of complex permittivity  $\varepsilon$  determine storage capacity and energy diffusive power respectively, however, energy losses are connected both to polarisation and insulation leakage current.

The measure of lag between dielectric polarisation and changes in electric field is the dielectric loss factor defined as:

$$\tan \delta = \frac{I_R}{I_C} = \frac{1}{\omega R C} = \frac{\varepsilon''}{\varepsilon'} \quad (12)$$

where the angle  $\delta$  is the cofactor of phase displacement  $\varphi$  to  $90^\circ$ .

Obtained through measurements quantities: impedance  $Z(\omega)$  and admittance  $Y(\omega)$  can be adopted as basic values enabling further processing of results of measurements in order to give an adequate account of given material properties which are of interest to us.

When research results are subject to interpretation through impedance spectroscopy, one has to bear in mind, that measured quantities are a measure of analysed system's properties, which is composed of electrodes and located between them material. Hence, the measured quantities give a picture of the entire circuit in sinusoidally alternating electric field, including lead resistance and inductance, stray capacitance and phenomena related to electrode polarisation.

Dielectric response of the composite non-woven PP / plasma layer is a function of both the physical structure of substrate and applied layer. So far, studies carried out by impedance spectroscopy allowed us to:

- identify the degree of porosity of the substrate on which the plasma layer was applied (Jaroszewski et al., 2010),
- determine the effect of surface resistivity of the composite relaxation processes (Pospieszna et al., 2010, Pospieszna & Jaroszewski, 2010),
- identify the strong dependence of dielectric composite properties upon a number of formed metal/metal-oxide layers (Ziaja & Jaroszewski, 2011).

## 5. Conclusions

Polypropylene in form of nonwoven fabric is promising material for EM shield composites. However, the use of this material is dependent on the possibility to cover it with another material exhibit conductive properties. This is possible only by using magnetron techniques.

The processes of formation of conductive bridges at the nonwoven fabric surface and on the fibres inside are critical to the screening factor. These processes can be examined both by analysing the surface morphology and dielectric properties.

## 6. Acknowledgment

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